Docket No.: 1422-0703PUS1
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF: Kazuhiro YOSHINO et al.	
Application No.: 10/560,239	Confirmation No.: 1480
Filed: December 12, 2005	Art Unit: 3761
For: ABSORBING MATERIAL AND ABSORPTIVE ARTICLE USING THE SAME	EXAMINER: WIEST, Philip R.

DECLARATION UNDER 37 C.F.R. 1.132

COMMISSIONER FOR PATENTS
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Masayoshi HANDA, residing in Hyogo-ken, Japan, hereby declare and state as follows:

1. I am thoroughly familiar with the contents of U.S. Application Serial No. 10/560,239 filed on December 12, 2005, entitled ABSORBING MATERIAL AND ABSORPTIVE ARTICLE USING THE SAME, its prosecution before the United States Patent and Trademark Office and the references cited therein.

2. I received a master's degree from Kanazawa University of Japan, faculty of natural science and technology in the year 1994, majoring in material chemistry.

3. I have been employed in Sumitomo Seika Chemicals Co., Ltd. in the year 1994 and have been assigned to the Research Laboratories.

4. I have been involved in the research and development of water-absorbent resin since 1998.

5. The following experiments were conducted by myself or under my direct supervision and control in order to (1) compare absorption capacity under no load in Wada (U.S. Patent No. 6150582) with water-retaining capacity of physiological saline in the present invention, and to (2) conduct a follow-up test of Water-absorbent resin (4) of Wada.

EXPERIMENTAL METHOD

1. COMPARISON OF WATER-RETAINING CAPACITY WITH ABSORPTION CAPACITY UNDER NO LOAD

The water-absorbent resins of the present invention and commercially available water-absorbent resins (named Samples A to D) that are recently obtained were evaluated for the absorption capacity under no load in accordance with Wada and the water-retaining capacity of physiological saline in accordance with the present specification to study the relationship therebetween. The measurement methods are as follows.

1-1. Absorption Capacity Under No Load (Wada)

First, 0.2 g of water-absorbent resin was uniformly placed into a nonwoven fabric-made bag (60 mm × 60 mm) and then immersed into an artificial urine (composition: an aqueous solution containing sodium sulfate of 0.2 wt %, potassium chloride of 0.2 wt %, magnesium chloride hexahydrate of

0.05 wt %, calcium chloride dihydrate of 0.025 wt %, ammonium dihydrogen phosphate of 0.085 wt %, and diammonium hydrogen phosphate of 0.015 wt %). Sixty minutes later, the bag was drawn up and then drained at 250 G for 3 minutes with a centrifuge, and the weight W_1 (g) of the bag was then measured. On the other hand, the same procedure was carried out using no water-absorbent resin, and the resultant weight W_0 (g) was measured. Thus, the absorption capacity (g/g) under no load was calculated from these weights W_1 and W_0 in accordance with the following equation:

Absorption capacity (g/g) under no load

= (Weight W_1 (g) - Weight W_0 (g))/Weight (g) of Water-Absorbent Resin

1-2. <u>Water-Retaining Capacity of Physiological Saline (The Present Invention)</u>

Two grams of a water-absorbent resin was placed in a cotton bag (Cottonbroad No. 60, width 100 mm × length 200 mm), and the cotton bag was placed in a 500 mL-beaker. Physiological saline was poured into the cotton bag in an amount of 500 g at a time, and the saline was dispersed so as not to generate a lump of the water-absorbent resin. The upper part of the cotton bag was tied up with a rubber band, and the cotton bag was allowed to stand for 1 hour, to sufficiently swell the water-absorbent resin. The cotton bag was spin-dried for 1 minute with a spin dryer (manufactured by Kokusan Enshinki Co., Ltd., H-122) set to have a centrifugal force of 167G, and a weight Wc (g) of the cotton bag containing swollen gels after the dehydration was determined. The same procedures were carried out without adding a water-absorbent resin, and an empty weight Wd (g) of the cotton bag upon wetting was determined. The water-retaining capacity was calculated in accordance with the following

formula:

[Water-Retaining Capacity of Physiological Saline (g/g)] = (Wc - Wd)/2

2. FOLLOW-UP EXPERIMENT OF WADA

2-1. REFERENTIAL EXAMPLE 4 (Corresponding to Water-absorbent resin (4)) OF WADA

2-1-1. Synthesis of Water-Absorbent Resin (Unless specified otherwise, half the scale of that disclosed in REFERENTIAL EXAMPLE 4 of Wada was used.)

A reaction solution was prepared by dissolving 3.235 g of polyethylene glycol diacrylate (average molar number of added ethylene oxide: 8) into 2,750 g of an aqueous solution of sodium acrylate with a neutralization ratio of 65 mol % (monomer concentration: 30 wt %). Next, this solution was degassed under a nitrogen gas atmosphere for 30 minutes, and then supplied into a reaction vessel as prepared by capping a stainless-steel-made double-arm type kneader of a capacity of 5 liters having two sigma type wings and a jacket. While maintaining the reaction solution at 30°C., the atmosphere inside the system was replaced with a nitrogen gas. Next, while the reaction solution was stirred, 0.955 g of 2,2'-azobis(2-amidinopropane) dihydrochloride, 0.48 g of sodium persulfate and 0.05 g of L-ascorbic acid were added, so that a polymerization reaction got started about 1 minute after. The polymerization was carried out at 30-80°C., and the resultant hydrogel polymer was separated out 60 minutes after the initiation of the polymerization.

The resultant hydrogel polymer had a finely divided diameter of about 5 mm. This finely divided hydrogel polymer was spread on a 50-mesh wire net and dried at 150°C. with hot air for 90 minutes. Then, the resultant dried product

was pulverized with a ball-mill and further classified with a wire net of 20 mesh, thus obtaining a formless pulverized water-absorbent resin precursor (A).

A surface-crosslinking agent comprising 0.3 g (1 part) of propylene glycol, 0.0075 g (0.025 parts) of ethylene glycol diglycidyl ether, 0.6 g (2 parts) of water, and 0.3 g (1 part) of isopropyl alcohol was mixed with 30 g (100 parts) of the resultant water-absorbent resin precursor (A). The mixture was mixed in a 1-L separable flask by spraying, while stirring with agitation blades. The resultant mixture was heated at 185°C. for 45 minutes, thus obtaining a water-absorbent resin (B), of which the average particle diameter was 420 μm.

2-1-2. Absorption Capacity Under No Load, Absorption Capacity Under Load, Amount of Water-Soluble Component, Absorption Speed, and Urine Resistance Index of Water-Absorbent Resin

The absorption capacity under no load of physiological saline, the absorption capacity under load, the amount of water-soluble component, the absorption speed, and the urine resistance index of the water-absorbent resin (B) were measured in accordance with the methods disclosed in Wada.

Measurement Apparatus for Absorption Capacity Under Load

As is shown in FIG. 1, the measurement apparatus comprises: a scale 1; a vessel 2 of a predetermined capacity as mounted on the scale 1; an air-inhaling pipe 3; an introducing tube 4; a glass filter 6; and a measurement part 5 as mounted on the glass filter 6. The vessel 2 has an opening part 2a on the top and an opening part 2b on the side. The air-inhaling pipe 3 is inserted in the opening part 2a, and the introducing tube 4 is fitted to the opening part 2b. In addition,

the vessel 2 contains a predetermined amount of artificial urine 11 (composition: an aqueous solution containing sodium sulfate of 0.2 wt %, potassium chloride of 0.2 wt %, magnesium chloride hexahydrate of 0.05 wt %, calcium chloride dihydrate of 0.025 wt %, ammonium dihydrogen phosphate of 0.085 wt %, and diammonium hydrogen phosphate of 0.015 wt %). The lower part of the air-inhaling pipe 3 is submerged in the artificial urine 11. The glass filter 6 is formed in a diameter of 70 mm. The vessel 2 and the glass filter 6 are connected to each other through the introducing tube 4. In addition, the upper part of the glass filter 6 is fixed so as to be located a little higher than the lower end of the air-inhaling pipe 3.

The measurement part 5 comprises: a filter paper 7; a supporting cylinder 8; a wire net 9 as attached to the bottom of the supporting cylinder 8; and a weight 10; and the measurement part 5 is formed by mounting the filter paper 7 and the supporting cylinder 8 (i.e. wire net 9) in this order on the glass filter 6 and further mounting the weight 10 inside the supporting cylinder 8, namely, on the wire net 9. The supporting cylinder 8 is formed in an inner diameter of 60 mm. The wire net 9 is made of stainless steel and formed in 400 mesh (mesh size: 38 μm). An arrangement is made such that a predetermined amount of water-absorbent resin 12 can uniformly be spread on the wire net 9. The weight 10 is adjusted in weight such that a load of 50 g/cm^2 can uniformly be applied to the wire net 9, namely, to the water-absorbent resin 12.

The absorption capacity under a load was measured with the measurement apparatus having the above-mentioned constitution. The measurement method is hereinafter explained. First, predetermined preparatory operations were made, in which, for example, a predetermined amount of the artificial urine 11 was placed

into the vessel 2, and the air-inhaling pipe 3 was inserted into the vessel 2. Next, the filter paper 7 was mounted on the glass filter 6. On the other hand, in parallel with these mounting operations, 0.9 g of water-absorbent resin 12 was uniformly spread inside the supporting cylinder, namely, on the wire net 9, and the weight 10 was put on the water-absorbent resin 12.

Next, the wire net 9, namely, the supporting cylinder 8 (in which the water-absorbent resin 12 and the weight 10 were put), was mounted on the filter paper 7. Then, weight W_2 (g) of the artificial urine 11, as absorbed by the water-absorbent resin 12 over a period of 60 minutes since the supporting cylinder 8 had been mounted on the filter paper 7, was measured with the scale 1. Then, the absorption capacity (g/g) under a load, at 60 minutes after the initiation of the absorption, was calculated from weight W_2 in accordance with the following equation:

Absorption capacity (g/g) under load

= Weight W_2 (g)/Weight (g) of Water-Absorbent Resin

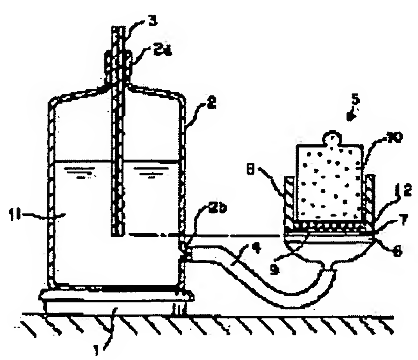


FIG. 1

Amount of Water-Soluble Component

First, 0.500 g of water-absorbent resin was dispersed into 1,000 ml of deionized water and stirred for 16 hours, and then filtered with filter paper. Next, 50 g of the resultant filtrate was placed into a 100 ml beaker, and 1 ml of a 0.1 N aqueous sodium hydroxide solution, 10.00 ml of an N/200 aqueous methyl glycol chitosan solution, and 4 drops of a 0.1 wt % aqueous Toluidine Blue solution were added to the filtrate. Next, the resultant solution in the beaker was subjected to colloid titration with an N/400 aqueous potassium polyvinyl sulfate solution to determine titration amount Y (ml) assuming that the moment at which the color of the solution had changed from blue to red purple was the terminal of the titration. In addition, titration amount Z (ml) was determined by carrying out blank titration in the same way as the above-mentioned, except that 50 g of the filtrate was replaced with 50 g of deionized water. Then, the amount of the water-soluble component (wt %) was calculated from titration amounts Y and Z and from neutralization ratio W (mol %) of the acrylic acid, as provided for the production of the water-absorbent resin, in accordance with the following equation:

Amount (% by Weight) of Water-Soluble Component

$$= (Z\,(\mathrm{ml}) - Y\,(\mathrm{ml})) \times 0.01 \times (72 \cdot (100 - W) + 94W) / 100$$

Absorption Speed

First, 50 g of physiological salt solution (0.9 wt % aqueous NaCl solution), as adjusted to 30°C., and a stirring rod are placed into a beaker of 100 ml, and then stirred at a rate of 600 rpm with a magnetic stirrer. Then, if 2 g of water-absorbent resin is added into the beaker, gelation occurs to decrease the fluidity,

and finally, the vortex of the stirring center disappears. A time, as spent from the addition of the sample till the disappearance of the vortex, was measured and regarded as the absorption speed.

Urine Resistance Index (Stability of Swollen Gel to Urine)

First, 2 g of water-absorbent resin was swollen to 25 times with an artificial urine containing L-ascorbic acid of 0.005 wt % (composition of the artificial urine: 95 g of urea, 40 g of sodium chloride, 5 g of magnesium sulfate, 5 g of calcium chloride, 4,855 g of ion-exchanged water) in a plastic vessel of 100 ml with a cap, and then this vessel was capped and left to stand stationary under an atmosphere with a temperature of 37°C. and a relative humidity of 90% for 16 hours. Then, a distance, where the surface of the gel layer ran on a wall face of the vessel in 1 minute after tilting the vessel at 90°, was measured and regarded as the urine resistance index. The longer the running distance is, the worse the stability of the swollen gel to urine is.

2-1-3. Water Absorption of Physiological Saline and Water-Retaining Capacity of Physiological Saline of Water-Absorbent Resin

The water-absorbent resin (B) was evaluated for the water absorption of physiological saline and the water-retaining capacity of physiological saline in accordance with the methods described in the present specification.

Water Absorption of Physiological Saline

In a 1000 mL beaker, 2 g of a water-absorbent resin was dispersed in 1000 g of physiological saline (0.9% by weight aqueous sodium chloride), and

the dispersion was gently stirred for 1 hour to sufficiently swell the resin. On the other hand, the physiological saline containing the swollen gel was filtered with a JIS standard sieve having an opening of 75 μm, the JIS standard sieve of which weight Wa (g) was previously determined. The filtered sieve was allowed to stand for 30 minutes in a state so that the sieve was tilted at a tilt angle of about 30 degrees to the horizontal to remove excess physiological saline from the water-absorbent resin. After a weight Wb (g) of the sieve containing the swollen gel was determined, the water absorption was calculated in accordance with the following formula:

[Water Absorption of Physiological Saline (g/g)] = (Wb - Wa)/2

Water-Retaining Capacity of Physiological Saline

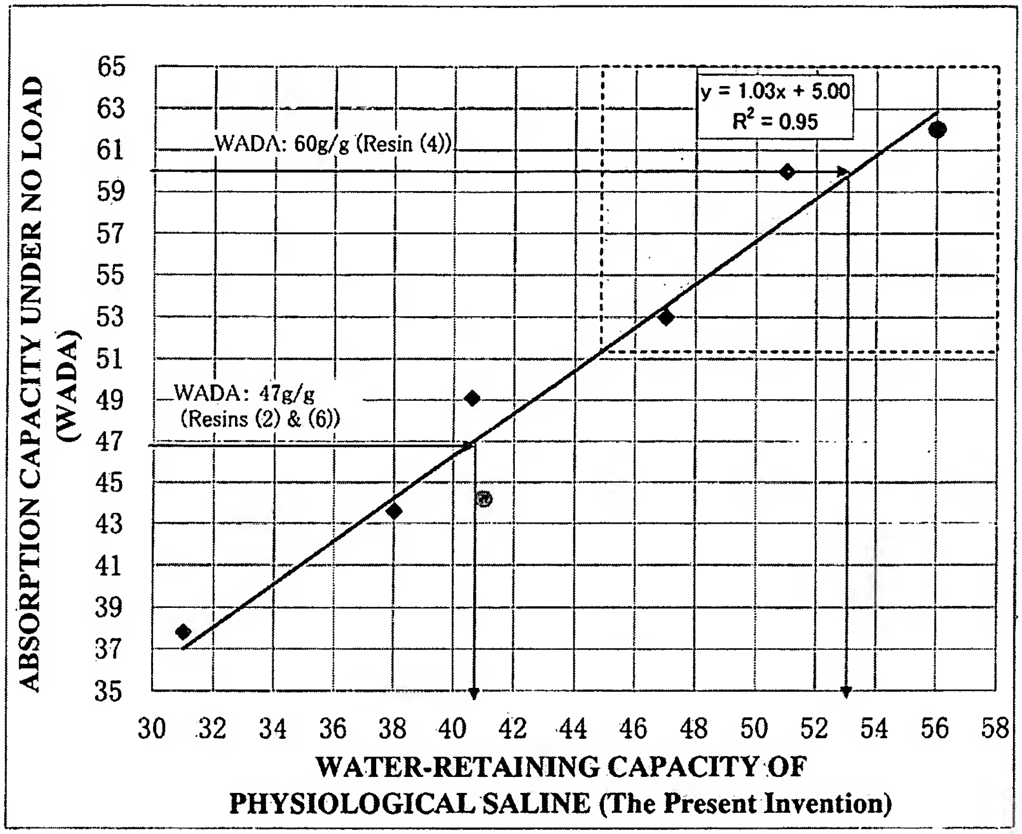
The method for evaluating the water-retaining capacity of physiological saline was carried out as described under item 1-2 of this Declaration.

3. MEASUREMENT RESULTS

3-1. Comparison of Water-Retaining Capacity with Absorption Capacity Under No Load

		Measurement Methods	
		Water-Retaining Capacity of Physiological Saline	Absorption Capacity Under No Load of Wada
Inventive Products	Production Example 1	41	44
	Production Example 3	56	62
Comparative Commercially Available Absorbent Resin	A	31	38
	B	38	44
	C	41	49
	D-1	47	53
	D-2	51	60
Remarks		Liquid: Physiological Saline, centrifugal force: 167 G × 1 minute	Liquid: Artifical Urine, centrifugal force: 250 G × 3 minutes

*: A, B, C, D are articles produced by diferent manufacturers.
D-1, D-2 show articles produced by the same manufacturer in different grade scale.



It can be seen from the measurement results for the water-absorbent resins of the present invention and the commercially available water-absorbent resins that both of the measurement values are well correlated. From the correlation, since the water-absorbent resins (2) and (6) of Wada have absorption capacity under no load of 47 g/g, a water-retaining capacity of physiological saline as measured by the method described in the present invention would be about 40 to 42 g/g or so, which are considered to be outside the claimed range of the present invention where the water-retaining capacity of physiological saline is 45 g/g or more.

3-2. Measurements of Properties of Water-Absorbent Resin (B)

From the results of 3-1., the synthesis of the water-absorbent resin (B) (corresponding to Water-absorbent resin (4)) having absorption capacity under no load of 60 g/g described in Wada was followed up, and whether or not the resulting water-absorbent resin (B) meets the requirements of the parameters mentioned in the present specification was confirmed. The measurement results are shown in the following table.

	Parameters of WADA					Parameters of the Present Invention	
	Absorption Capacity Under No Load	Absorption Capacity Under Load	Amount of Water-Soluble Component	Absorption Speed	Urine Resistance Index	Water-Retaining Capacity	Water Absorption
Water-Absorbent Resin (B)	38	26	7	54	0	29	46
Values Disclosed in Wada	60	13	13	48	0	-	-

As shown in the above results, the water-absorbent resin (B) synthesized in accordance with Wada was found not to have the properties of the absorption capacity under no load as disclosed in Wada, i.e., 38 vs. 60. Since the numerical figures on other properties, such as absorption capacity under load, the amount of water-soluble component, the absorption speed, and the urine resistance index of the water-absorbent resin (B) fairly approximate those disclosed in Wada, it is assumed that there should be some sort of typographical errors in the absorption capacity under no load (namely, the value therefor should have been far lower than 60 g/g).

Statement Under 18 U.S.C. § 1001

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Dated: June 3, 2008 By Masayoshi Handa

Masayoshi HANDA